



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS:        Miroslav Chmelir et al.  
Application NO.:    09/554,090  
FILED:                14 June, 2000  
FOR:                  METHOD FOR PRODUCING WATER-SOLUBLE OR WATER-  
                             SWELLABLE POLYMERIZATES WITH A VERY LOW  
                             RESIDUAL MONOMER CONTENT, PRODUCTS PRODUCED  
                             ACCORDING TO THIS METHOD AND THE USE THEREOF

DECLARATION UNDER 37 C.F.R. § 1.132

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

I, Dr. Gregor Herth, hereby declare as follows:

1.        I am a citizen of Germany, residing at 47799 Krefeld, Sprödenalstr. 82
2.        I studied chemistry at the University of Marburg and received a degree in the field of Polymer Chemistry in the year 1998.
3.        Since 1998 I have been employed as a chemist in the field of research and development of polyacrylamides and I am still working in this field for the company of Stockhausen GmbH, Krefeld, Germany.
4.        The following tests were made under my supervision and control:



**I. Test according to the US application No. 09/554,090**

**Example**

First, 602 g of acrylic acid was mixed with 550 g water in a polymerisation vessel. Then the monomer solution was neutralized with 692.7 g of ammonia (25%) to a neutralization level of 60%. The monomer solution was then cooled to 10 °C and purged with nitrogen. Following addition of the initiator solutions (2.0 g of sodium peroxodisulphate, 0.4 g of 2,2'-azobis(2-methyl-propionamidine) dihydrochloride, each in 20 ml of water, and 0.1 g Irgacure® 651 in 2 g of acrylic acid) the polymerisation was initiated using UV light. The maximum temperature of 102 °C was reached within 12 minutes. After polymerization had ended, the polymer was heated at 140 °C for different times to show the influence of heating time on residual monomer content of acrylic acid and gel content.

**II. Measurement of gel content**

0.8 g of the polymer sample is weighed to an accuracy of 0.01 g into a 1000 ml beaker. 800 ml of tap water is added in such a way that no lumps are formed. Then the solution is stirred for 90 minutes with the finger stirrer at 300 r.p.m.. When stirring is finished the solution is placed on a standard sieve (315 µm) and once it has passed through the sieve this is rinsed through five times with one liter of fresh water from the beaker. After the rinsing water has passed through the sieve any residual water under the sieve is wiped off with the rubber policeman.

Using the rubber policeman and the powder funnel the residue is transferred to a 100 ml measuring cylinder and the volume determined.

### III. Results

Heating time at 140°C [min]	Residual monomer acrylic acid [ppm]	Gel content (0.1% sol.) [ml/l]
5	11700	15
30	130	27
45	53	36
60	16	40
90	20	43
150	9	71

As can be seen from the results of the heat treatment the residual monomer content of the polymer is reduced with increasing heating time. Another important property for water soluble polymers is the gel content. The gel content is formed by unintended crosslinking of the polymer during heat treatment. Gel content represents undissolved polymer i.e. the lower the gel content the better is the performance of the polymer as functional additive, e.g. as flocculation polymer in paper processing. In order to reach a low residual monomer content and to avoid a higher gel content it is recommended to limit the heating time period between 10 minutes and 2 hours.

All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true, and further, these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the patent application or any patent issued thereon.

28. 04. 2005

(Date)

y. Herth

(Dr. Herth)